

## ART. XXXVII.—*Flotation of Minerals.*

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### Introduction.

The various flotation concentration processes have in the last few years attained much prominence in Australia, and particularly at Broken Hill, and promise to displace many of the ordinary gravity processes in other parts. Very little literature has appeared on this subject, and, with the exception of a few scientific papers, most of it has been descriptive of the working of various processes, and not on the investigation of the general underlying principles. The two usual explanations given as to the cause of the flotation of minerals and metals in various solutions are:—

- (1) That it is due to certain surface tension phenomena.
- (2) That it is due to the attachment of certain gases to the minerals which lift the particles to the surface.

In a paper by J. Swinburne and G. Rudolf,<sup>1</sup> the authors explain why the sulphides rise in preference to the silicates, etc., and why the bubbles remain attached to the sulphides, as due to the combined effects of surface-tension, cohesion and adhesion. They consider that the rise of temperature to near boiling point is necessary for flotation for the same reason.

### Behaviour of Minerals and Metals in Water.

The object of the writer's work was to investigate the attachment of gases to the different minerals and metals, and incidentally to study the adhesion, or wetting of different minerals and metals, and their subsequent flotation.

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<sup>1</sup> The Physics of Ore Flotation. Chemical News, 29th December, 1905.

*Cold water.*—Minerals and other substances differ in their inclination or otherwise to become wetted by water or by other liquids. Many minerals when dropped carefully on to the surface of water remain on it and do not sink. Some at once sink to the bottom. Particles of considerable size may be floated in this manner, and sometimes considerable force is required to make them sink. Some minerals, when ground so as to pass a 30 mesh sieve, form a film on the surface of the water, and this film will support a considerable weight. A certain sample of zinc blende concentrates could be heaped up in this manner to a depth of  $\frac{1}{4}$  inch on the surface of water in a 50 c.c. cylindrical beaker. The weight of material was 15 gms., and the top portion was quite dry. The following is a list of the minerals tried that would float on the surface of water:—Galena, zinc blende, sulphur, azurite, malachite, rhodonite, garnet, calcite, mica, telluride of gold, pyrite, pyrrhotite, wolfram, cassiterite, serpentine. And besides this all the metals in the form of foil would easily float. Some samples of quartz will float and others will not. Particles of glass and turquoise would not float.

The weight of each particle on the surface causes a distinct depression or sag round itself, and the apparent attraction of one particle for another is probably due to the depressed surface caused by this sagging. If some liquids (such as alcohol in the form of a drop on a glass rod) are brought near the particles floating on the surface, the particles are repelled from the rod. If alcohol is added to the water the particles become wetted and sink. Caustic soda acts similarly. This is apparently a surface tension phenomenon. In the case of alcohol on the glass rod, the vapour of the alcohol mixing with the water decreases the surface tension and increases the adhesion of the water for the mineral. The water wets the particle at a higher level on the side nearest the glass rod; an inclined plane is formed on the side furthest from the rod, and the particle is apparently repelled. Water has the greatest surface tension of any liquid under ordinary atmospheric conditions except mercury, and thus, if another liquid is added to it, the surface tension is decreased. If the minerals in the above list have been previously wetted they will not float on the surface until they are

dried or partially dried. If some ground mineral is wetted and covered with water, and then is exposed to the air by causing the water to run to one side of the containing vessel and the water is then brought carefully back, some of the mineral will be seen to float on the surface of the water. This form of flotation takes place to a greater or lesser extent with most minerals, and is apparently a different phenomenon from the simple floating of particles by dropping them on the surface. In the latter case there is an almost continuous film of air or gas surrounding the under side of the mineral, and by far the greater portion of it is projecting out of the surface of the water. That there is not a continuous film, and that the particle is partly wetted can be shown by floating some magnetic substance, as pyrrhotite, or a piece of iron wire, and attracting it with a magnet, when the surface of the water will be seen to have been dragged up with the mineral or wire. When the substance is finally lifted out of the water, water is carried up on to the magnet. In the case of the wetted mineral being floated by being brought into contact with the air, the particles will be seen to be almost entirely submerged, only a portion like a pin's point being above the surface, and, although a distinct depression of the surface is caused, it is not so apparent as in the other case.

When the minerals are made to sink they apparently carry a bubble of some gas down with them. This bubble can be seen as a rule on most particles. When the finer-grained material is heaped up on the surface (as in the case of the zinc concentrates referred to) till the weight is too great, the whole mass causes the surface of the water to sag; and finally to break through, and carries a large amount of air down with it, the inside of the envelope being quite dry.

*Hot water.*—In hot water the behaviour of the minerals and metals is different. Most of them will float but for a few seconds only, becoming wetted and sinking, usually a bubble of gas leaving each particle as it sinks. The fine grained material can be heaped up similarly, as with cold water, a large number of bubbles being observed on the under surface of the mass.

*Under the surface of water.*

When the minerals are submerged in water and the water is heated, many of them will float either wholly or partially. Sulphur, crystalline azurite and malachite, some galenas, some blendes, some chalcopyrites and stibnites, and some of the metals, copper being the most persistent, will float in water on heating. A sample of zinc concentrates from the Potter process (floated with  $\text{H}_2\text{SO}_4$ ) floated when heated in water, and when boiled the "scum" continued indefinitely. If boiled violently and continuously, the concentrates will sink, but if allowed to cool will again float on heating. This was repeated twelve times, and would apparently continue indefinitely, if after each time the water was allowed to cool for some time. Air will get into boiling water either being dissolved or being carried down mechanically as bubbles, as the following will show. A large beaker of distilled water was boiled continuously for two hours. An inverted funnel was then placed in the beaker and an inverted burette filled with the boiled water was placed with the wide end fitting over the stem of the funnel. Air was then slowly collected in the burette by the bubbles coming under the funnel and displacing some of the water in the burette. The funnel was arranged so that it did not rest on the bottom of the beaker, but allowed the water to circulate under it. In some cases the minerals appear to evolve a gas, and usually the water begins to boil and water vapour forms first on the minerals, or substances introduced into it, the latter giving a starting point apparently preferable to the sides of the containing beaker.

A sample of zinc concentrates under water was heated in a 10 c.c. graduated pipette and the expansion observed. These concentrates were from a Potter (acid) float, washed and dried, and then introduced into the pipette, which was immersed in a large beaker of water, and this water was then heated to boiling. The volume of concentrates as measured in the pipette was 2 c.c., and the surface of the water above them was 5.9 c.c. The greatest expansion noted by taking the difference in the reading of the level of the top of the water was 1.3 c.c. The expansion noted by the difference in the levels of the top of the concentrates before and after heating was 0.8 c.c. The volume

begins to increase at about 82 deg. C. up to 100 deg., the range of temperature at which flotation usually takes place. Just before the water began to boil some of the concentrates began to float and the reading could not be taken.

*Behaviour under reduced pressure.*

Many substances when sunk in water will rise to the surface when the pressure above the surface of the water is lowered. Among the substances tried the following floated:—All the metals in the form of foil, naphthalene, sulphur, graphite, mica, and zinc blende concentrates. Particles of galena, zinc blende, cerussite, river sand, and calcite came to the surface, but sank again. When a mixture of particles of zinc blende and river sand was put into a vacuum flask under water under reduced pressure, it was noticed that the zinc blende came up more persistently than the sand, which rose to the surface three or four times, and would not do so again. The particles of mineral are caused to float by gas bubbles attached to them carrying them up to the surface. In some cases these bubbles are strongly attached to the minerals. A burette was taken and filled with mercury to within about an inch of the top, and about 1 c.c. water was added on top of the mercury. A few particles of different minerals were dropped through the water on to the mercury, each having a bubble attached to it. A rubber cap with a clip was then put over the end of the burette and the lower end was connected up with a water pump. As the level of the mercury was lowered the bubbles increased in volume, and remained on the particles till the level of the mercury was lowered considerably. Some zinc concentrates from an acid float were immersed in water in a 10 c.c. graduated pipette, and the expansion noted when the pressure was reduced above the surface of the water. The greatest expansion noted before the mineral began to float was 0.8 c.c., the original volume being 2 c.c.

On some minerals bubbles will form, when subjected to reduced pressure, under water, but the minerals do not float, as quartz, garnet, gypsum, haematite, wolfram, cassiterite and glass. Pieces of metal in the form of foil will float even after

being repeatedly sunk. If the water is boiled free from air the metals will not float when the first bubbles attaching themselves are removed. A piece of copper foil, which was immersed in air-free water, and which would not float when the pressure was reduced, floated on the surface when brought in contact with the vapour above the surface of the water. Crystalline pieces of the following minerals under water under reduced pressure were all coated with bubbles:—Galena, zinc blende, garnet tourmaline, pyrite, gypsum, ardashite, calcite, cerussite, and quartz. These minerals were then placed in water, boiled free from air, and again became coated with bubbles on reducing the pressure, thus showing that some gas is carried down with each mineral as it sinks. The metals had also bubbles attached to them, but in freeing these with a glass rod only a few very minute ones formed again. After the bubbles have formed on these minerals, if atmospheric pressure is restored, the bubbles become almost invisible, but form again in the same position on again lowering the pressure. Tarnished minerals form as many bubbles on their surfaces as the clean faced ones.

## Behaviour of Minerals and Metals in Dilute Acid Solution.

### *On the surface.*

*Cold.*—Similar to water even with strong  $\text{H}_2\text{SO}_4$ .

*Hot.*—None of the minerals would float when sprinkled on the surface of a hot  $\text{H}_2\text{SO}_4$  solution even when very dilute (1 per cent.) becoming at once wetted and sinking. Copper foil will float on a hot 3 per cent.  $\text{H}_2\text{SO}_4$  solution. The sample of zinc concentrates forms a film the thickness of one particle only, the remainder falling through the surface, but coming up again in the form of agglomerated masses.

*Heated in acid solutions.*—Most of the sulphides will float if heated in a weak  $\text{H}_2\text{SO}_4$  solution. Some samples of pure galena and zinc blende would not float, but on the addition of calcite and generation of  $\text{CO}_2$  floated. Haematite, biotite, quartz, wolfram, cassiterite, rhodonite, calcite, siderite, musco-

vite, serpentine would not float. In the case of the sulphides  $\text{H}_2\text{S}$  is given off to a greater or lesser extent in every case. A sample of copper matte would not float in acid solution. To show the amount of acid required a small test with a sample of the South Mine, Broken Hill, tailings was carried out, taking 10 gms. of the tailings, and 50 c.c. water, and adding different amounts of acid and heating to about 95 deg. C.

With a .36 per cent.  $\text{H}_2\text{SO}_4$  solution no float took place.

With a .72 per cent.  $\text{H}_2\text{SO}_4$  solution a small scum formed.

With a .9 per cent.  $\text{H}_2\text{SO}_4$  solution a poor float was obtained.

With a 1.4 per cent.  $\text{H}_2\text{SO}_4$  solution a good float was obtained.

These tailings contain much calcite, and therefore use up more acid than most of the other companies' tailings.

*Under reduced pressure.*—The same lot of crystalline particles tried with water alone were tried under reduced pressure in an acid solution ranging from 1 up to 10 per cent.  $\text{H}_2\text{SO}_4$ . The bubbles formed as before, but were of a different nature, appearing to be larger, and coming away from the minerals more readily. Fewer bubbles were on the quartz and gypsum than on the other minerals. In an air-free, dilute  $\text{H}_2\text{SO}_4$  solution, on fragments of the various minerals being dropped in bubbles form on their surface. In the case of quartz and rhodonite and the other silicates a few bubbles were carried down, but on shaking these off no more formed. This also was the case with copper foil. Bubbles formed on the sulphides, garnet, and tourmaline even after those first formed were detached. Some cubical fragments of galena were coated with lead chromate and sulphate respectively and immersed in an air-free acid solution, under reduced pressure. Only a very few bubbles formed on these particles. Most of the sulphides will not float in the cold with dilute acid. The zinc blende in a sample of South Mine tailings kept coming up to the surface, but fell back again. The carbonates are attacked by very dilute acid solutions under reduced pressure. It is interesting to note that siderite and magnesite are attacked by cold acid under reduced pressure.

Some Broken Hill Proprietary's zinc concentrates, washed free from soluble salts, were boiled in a 2 per cent.  $\text{H}_2\text{SO}_4$  solution. The following metals went into solution, Fe, Al, Zn, Mn, and  $\text{H}_2\text{S}$  was given off. A sample of the working solution from this



company's process contained  $\text{SiO}_2$  (0.112 gms. per 100 c.c.), Fe, Al, Zn, Mn, and Ca. A sample of the Central Mine's concentrates (oil and acid process) was washed with strong NaOH, and then washed till free from NaOH. The zinc blende would not then float on top of air-free water on boiling, but on making acid with  $\text{H}_2\text{SO}_4$  a float was obtained. No float could be obtained by adding ammonium carbonate. Another sample of the Proprietary's zinc concentrates washed free from acid, and then washed with strong NaOH, and then washed free from alkali would not float on heating in tap or air free water, but floated on adding acetic or sulphuric acid.

### Behaviour of Minerals and Metals with Oils, Etc.

*On the surface.*—None of the minerals tried would float on the surface of oleic acid (commercial), kerosene, or alcohol. Copper foil floated on oleic acid, but not so persistently on kerosene. Iron wire would not float on kerosene or oleic acid, but showed a tendency to float, not sinking immediately. Zinc blende, galena and the other minerals would not float on alcohol but did not immediately sink.

*On heating.*—None of the minerals floated on heating in kerosene or oleic acid.

*Under reduced pressure.*—None of the minerals or metals floated when immersed in oleic acid, alcohol, or kerosene under reduced pressure.

### Behaviour of Oiled Minerals in Water and Acid Solutions.

Nearly all the minerals and metals will absorb oil if shaken up in water to which oil has been added. If previously oiled as with oleic acid, vaseline, or kerosene, nearly all minerals will float in water. Some float in the cold; others require heating or reduced pressure. The following float well:—Calcite, sulphur, galena, garnet, zinc blende, wolfram, cassiterite, siderite, cerussite, magnesite, azurite, malachite, rhodonite, and some quartz, and the metals in form of foil. The following give no



float, or a very poor float:—Muscovite, amorphous malachite, turquoise, gypsum, quartz and particles of slate. Calcite and muscovite if crushed together can be separated by oleic acid, the calcite floating, and the muscovite not absorbing the oil. Copper matte will give a flotation with oleic acid in  $\text{H}_2\text{SO}_4$  solution. Whether a mineral is caused to float, or is merely collected together in a mass or in small rounded balls, or is made granular but coherent, is dependent on the quantity of oil used. A series of tests carried out showed that with an excess of oil over the mineral the latter was caused to float by being carried to the surface of the water by the lighter oil. With oil and the mineral in certain ratios as 5 gms. oil to 20 or 30 gms. of zinc blende the oil and mineral formed one globular coherent mass. By increasing the amount of mineral and keeping the same amount of oil, smaller rounded masses of oil and mineral formed which would not float until finally with a large amount of mineral (150 gms.) and 5 gms. oil a granular product was formed which would float on violent agitation, being buoyed up by attached air bubbles. On adding acid to the minerals which have absorbed oils in presence of water, most minerals other than sulphides and metals will not float on heating, the acid apparently causing them to separate from the oil. The amount of acid required to make the action selective is very small, but from the results of numerous tests it is apparent that with only minute quantities of acid the action is only partially selective. If more acid is used the freer from gangue the concentrates will be. Calcite and oleic acid will not mix in the presence of oxalic acid, although the calcite is not apparently attacked. Calcite will absorb oleic acid in the presence of acetic and lactic acids. There is no very marked difference between the behaviour of sulphur and oleic acid in an acid solution or in water. Carbon bisulphide acts similarly to an oil, and is apparently absorbed by the minerals.

*Oiled Minerals under reduced pressure.*

Crystalline fragments of tourmaline, garnet, zinc blende, galena, pyrite, quartz, gypsum, andalusite and calcite—were taken, two of each, one oiled with oleic acid and the other

unoiled. These were immersed in water, and the pressure reduced above the water. Bubbles immediately formed on the oiled minerals, and on further decreasing the pressure, bubbles larger and in greater number formed on the oiled than on the unoiled minerals. The nature of the bubbles on the oiled and unoiled minerals were different, those on the oiled being of an inverted watch glass shape when first formed. On adding acid ( $\text{H}_2\text{SO}_4$ ) the bubbles change in character. Most of the bubbles left the unoiled quartz and gypsum, but not the other minerals. At the lower pressure  $\text{CO}_2$  was evolved from the calcite which was not attacked at first, showing that the acid solution gets under the oil film on the mineral. With a lens minute specks could be seen on the faces of the pyrite crystal which gradually grew into larger bubbles on reducing the pressure. On restoring atmospheric pressure the bubbles became almost invisible again. The bubbles on the oiled minerals were of a more permanent character and the manner of leaving the minerals was also different. In the case of the oiled minerals the bubbles become distorted and drag away more than in the case of the unoiled minerals. On warming the solution the bubbles clung much more tenaciously to the sulphides than to the other minerals, the quartz, calcite, and gypsum being almost free from bubbles.

If a layer of oil is floated on top of water and a particle of a mineral as galena or quartz is dropped on to it, the mineral will only sink as far as the top of the water and remain there. Minerals will sink through alternate layers of different oils floating on water, and will remain floating on the top of the water. If a particle of some mineral is sunk in water and a layer of oil is floated on top of the water, and if the pressure is reduced above the surface of the oil, the mineral particle will rise as far as the surface of the water, and will not penetrate the oil layer. Three beakers were taken, each containing a saturated  $\text{CO}_2$  solution in water. A layer of oleic acid was added to form a layer over the surface of one, kerosene in another and petrol in the third. Particles of different minerals were then dropped into the beakers, and their behaviour noted. All the minerals carried down oil with them in the form of a circular "blob."  $\text{CO}_2$  gas immediately began

to collect under the surface of the oiled particles in the form of very small bubbles, which collected to form one large bubble, until on becoming too large it broke away and came to the surface, the oil returning to its original form until another large bubble formed from the smaller bubbles collecting together. On adding  $\text{H}_2\text{SO}_4$  many bubbles and apparently the excess of the oil left the minerals, and on standing or on heating, the minerals, quartz, calcite, and gypsum became practically free from bubbles. The sulphides, metals and garnet still had the bubbles collected on them, but in a different form, each bubble being separate, and not combining with the others to form one large bubble under the surface of the oil as was the case with water alone.

A very small amount of oil only is required to oil a considerable amount of any mineral. Some South Mine tailings weighing 20 gms. was added to water, and 1 drop (.026 gms.) oleic acid added and the mixture shaken up, and put in a vacuum flask. On reducing the pressure the whole product, metallic and non-metallic, floated to the surface. With 40 gms. only about half was raised.

### In Alkaline Solutions.

*On the surface.*—Most minerals will float on the surface of an NaOH solution, but for a few moments only, and then become wetted and sink.

*On heating.*—When heated in a solution of NaOH, the minerals will not float. On adding an ammonium salt, although gas is given off, no float takes place. With some zinc concentrates that have been previously floated, a good float was obtained in an ammonium carbonate solution. With  $\text{NH}_4\text{Cl}$  a partial and non-selective float was caused. Oleic acid saponified with ammonia will cause non-selective flotation, and on adding  $\text{H}_2\text{SO}_4$  till acid, the silicates will sink, and the sulphides remain floating. A sample of zinc concentrates would not float in NaOH, and on addition of  $\text{H}_2\text{SO}_4$  till acid would only float after heating for a considerable time.

*Under reduced pressure.*—Bubbles formed on the minerals under an NaOH solution under reduced pressure, the solution

being free from air. On making acid with  $\text{H}_2\text{SO}_4$  very few bubbles remained on any of the minerals, but more were on the sulphides than the quartz and silicates.

### Behaviour of Minerals and Metals in saturated Solutions of Gases.

*In water saturated with  $\text{CO}_2$ .*—Copper foil and other metals and also all the minerals constituting the Broken Hill tailings will float in water saturated with  $\text{CO}_2$ , being brought to the surface repeatedly by the  $\text{CO}_2$  bubbles. Bubbles form on all the minerals, and are not confined to the sulphides. Very few form on gypsum and fewer on the quartz than on the sulphides. On addition of  $\text{H}_2\text{SO}_4$  no bubbles remained on the gypsum and very few on the quartz, the garnet still having bubbles attached to it. The action is similar with a saturated solution of  $\text{SO}_2$ . Hydrogen peroxide is also similar in its action to the saturated solutions. The manner in which bubbles of gas are formed in a solution is very important as regards their subsequent attachment to the minerals. A very rapid current of bubbles may be evolved, but the bubbles may not attach themselves to mineral particles, and thus may not be of any use in their subsequent flotation. Sometimes the rapid evolution of a gas is useful as in the case where flocculent masses of the sulphides are held together by gas bubbles insufficient to carry them to the surface, and the rush of bubbles to the surface getting underneath these agglomerated sulphides causes them to be carried to the surface. The condition that seems to be the best for the attachment of the gas bubbles to minerals and metals is when a saturated solution is slowly evolving bubbles of gas that are being thrown out of solution by rise of temperature, or lowering of pressure. This takes place to a certain extent in all the flotation processes where a hot solution is used, the dissolved air,  $\text{CO}_2$ ,  $\text{H}_2\text{S}$  and any other gases in solution being driven out when the temperature is raised. In the case of saturated solution of gas, bubbles will form on almost anything introduced into the solution. A common example is the formation of bubbles on pieces of cork in soda water and on the sides

of a glass. On addition of acid the bubbles have a tendency to select the sulphides on which to form. Some zinc concentrates obtained from Central Mine tailings, Broken Hill, by a  $\text{H}_2\text{SO}_4$  float were washed well, dried and then floated in water with atmospheres of different gases above the water.

The following arrangement of apparatus was used:—

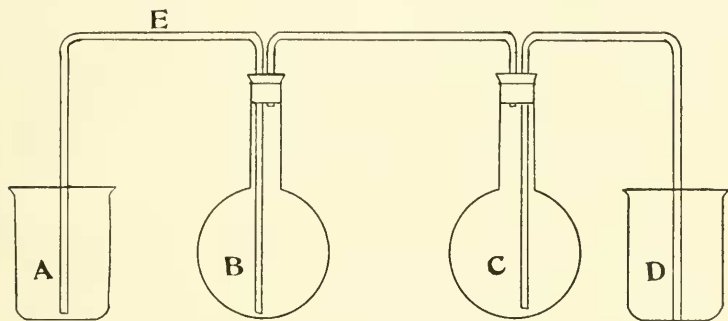


Fig.A

The zinc concentrates were placed in flask B, and boiled for a considerable time till the concentrates sank to the bottom. The flask was then filled to the top with boiling water. Flask C was filled with the gas to be used. The beaker D contained a saturated solution of the gas used, and the beaker A was half filled with air-free water. Some of the water in B was then syphoned into A, the space being taken up with the gas from C. B was then boiled, and shaken up, after closing the clamp E.

The gases tried were  $\text{H}_2\text{S}$ , nitrogen,  $\text{CO}_2$ ,  $\text{SO}_2$ , oxygen, hydrogen, water vapour and air. Flotation took place in every case, the scum formed in the case of  $\text{H}_2\text{S}$  having a slightly different appearance from the others. This treatment was equivalent to boiling the sulphide in a saturated solution of the gases named, and at the same time preventing all the gas being driven off before the solution became hot.

### The Action of Chlorine.

If particles of minerals are dropped into chlorine water, bubbles form on the sulphides if oiled or unoled, fewer forming on quartz and other silicates. Under reduced pressure in chlorine water the galena and zinc blende are thickly coated with bubbles, the galena being attacked by the chlorine, since the faces rapidly tarnish. Very few bubbles come off the quartz and pyrite. A sample of Broken Hill Proprietary zinc concentrates was washed till free from soluble salts, and stood for half an hour with a dilute chlorine water solution. On analysing the solution it was found that considerable amounts of Pb, Fe, and Zn, and a lesser amount of Al had gone into solution.  $\text{SO}_4$  was present in the solution, but no free chlorine. With these metals going into solution free sulphur must be liberated to some extent. If a sample of Broken Hill tailings is washed with Cl water it is noticed that the sulphide particles have a tendency to agglomerate and become buoyant, and are thus well prepared for subsequent flotation either by film flotation or otherwise. If a sample of tailings is shaken up with Cl water, and then a minute amount of some light oil, as petrol or gasolene, is added with a little  $\text{H}_2\text{SO}_4$ , a good flotation takes place in the cold if calcite is present to evolve  $\text{CO}_2$ . If instead of adding acid, water saturated with carbon dioxide is added, the same result is obtained. On some tailings a good flotation with Cl water and  $\text{H}_2\text{SO}_4$  is obtained in the cold without the addition of any oil.

### Film Flotation.

Film flotation is the term used to describe flotation when the mineral separated floats on the surface of the liquid to the depth of the thickness of one particle only, and not as a coherent scum as in the case of the Potter and other processes. In film processes, as the De Bavay, the sulphides, after being wetted, are brought into contact with the air, and fed on to the surface of water, and remain floating as a film and not as a scum. A sample of zinc blende obtained by film flotation

(using  $\text{H}_2\text{SO}_4$  and oleic acid) was immersed in boiled water and boiled under reduced pressure for three hours, as much of the air being pumped out as possible. All the mineral was then on the bottom of the flask. The top of the flask was then closed. On being brought into contact with the vapour above the water, a film was again formed. When examined with the aid of a lens the film was seen to be almost entirely submerged, only fine points of the particles projecting above the surface. A film of magnetic sulphide of iron on the surface of water when attracted by a magnet drew up the surface of the water with the mineral. Copper foil will not float in air-free water under reduced pressure, but if allowed to come in contact with the vapour above the water will float, similarly to the film float obtained with minerals.

Another sample of zinc concentrates from a film float was floated on the surface of water in a flask under reduced pressure, and exhausted as far as possible by a water pump. Most of the particles forming the film sank, although the remainder persistently floated. With a lens bubbles could still be seen attached. It was frequently noticed that under reduced pressure some mineral with oil would rise to the surface in agglomerated masses, and then spread out to form a film on the surface. Sometimes a mass of sulphides clinging to a bubble on coming to the surface and the bubble bursting, the sulphides would spread out as a film, the free surface of the water taking the place of the surface of the bubble.

### **Action of Acids and Oils in Film Flotation.**

The ores used in the following tests were freshly broken and tested as soon as crushed, so as not to allow the surface of the minerals to become altered. They were all crushed through a 40-mesh sieve, and flotation as a film was tried in water alone, with oil, and with oil, acid and water.



TABLE A.

Sample	With water alone	Oil and water	Acid, oil and water
1—Glassford Creek grey copper ore and garnet gangue	Non-selective	Non-selective	No float at all at first, but a non-selective float on long exposure to air
2—Queen Bee chalcop- pyrite with slate gangue	Tends to be selective	Selective till almost all the sulphide has been floated, then gangue be- gins to float	Selective
3—Mount Morgan pyrite and chalcopyrite	Tends to be selective	Similar to Queen Bee	Selective
4—Junction North ore (Broken Hill)	Non-selective	Non-selective	Selective

### Effect of Tarnishing the surface of Sulphides.

If the surfaces of the sulphides are tarnished, as a rule these sulphides will not float. Taking the Horwood process as an example, the mixture of lead and zinc sulphides is given a slight roast, which is sufficient to cause the galena to become coated with lead sulphate. The zinc blende is not much altered at the low temperature at which the roasting is carried out. When the material is then heated with an oil and acid solution only the zinc sulphide floats. If galena is coated by treating with strong  $\text{H}_2\text{SO}_4$  it has a tendency to prevent it floating; chromic acid has the same effect. Chlorine and nitric acid, which rapidly tarnish the surface of galena, do not in any way prevent flotation, but on the other hand have a beneficial effect. This may be due to the liberation of free sulphur (which does take place) on the surface of the sulphide, sulphur being one of the most easily floated substances. Some ores that will not float in their natural condition will float after being heated, the slight calcining having a beneficial effect on the flotation. This is noticeable with some of the heavy Tasmanian zinc-lead ores.

### Specific Gravity of Floated Materials.

The specific gravity of some zinc concentrates from a Potter (acid) float was found to be 2.81. The specific gravity of zinc blende is usually about 4. Two other samples of zinc concentrates from a process in which oil was used had a sp. gr. of 2.55 and 2.61 respectively. The low sp. gr. noted was due to absorbed gases.

A sample of some clean crystalline galena was crushed and sifted, and 20 gms. of the material remaining on each sized sieve was taken and added to 100 c.c. water. Half a gm. of oleic acid was then added, and well shaken up with the galena. The specific gravity of each sized mineral was then taken, and resulted as follows:—

	Spc. Gr.
On 40 sieve ... ..	6.4
On 60 sieve ... ..	6.1
On 80 sieve ... ..	6.2
On 100 sieve ... ..	5.8
On 180 sieve ... ..	5.8
Through 180 sieve (partial float) ... ..	3.9

Some of the finest grained material began to float on violent shaking. The specific gravity of galena is 7.4.

### The Adsorption of Gases by Minerals.

To ascertain if oxygen was absorbed from the air by an acid and oil solution with minerals, the following test was carried out:—Fifty gm. zinc concentrates were added to 200 c.c. water, and then 2 c.c.  $\text{H}_2\text{SO}_4$  and 0.5 c.c. oleic acid was added. This solution was agitated for  $\frac{1}{4}$  hour, and allowed to stand 18 hours in an airtight vessel. An analysis of the gas above the solution showed that it was composed of 14 per cent.  $\text{CO}_2$ , 18 per cent. oxygen, and 68 per cent. nitrogen. No  $\text{H}_2\text{S}$  could be detected. Blank tests were made with (1) acid, and (2) oil in the same proportion as above, the gas above the solution in each case having the composition of atmospheric air. Another test with clean galena from Broken Hill, with the

same amount of oil, acid and water, agitated for  $\frac{1}{4}$  hour, showed that the gas above the solution consisted of—

1 p.c. (absorbed as  $\text{CO}_2$ ).

21 p.c. oxygen.

78 p.c. nitrogen.

Lead acetate paper showed a reaction for  $\text{H}_2\text{S}$ .

With mixed magnetite and zinc blende, Mt. Garnet ore (Queensland)  $\text{H}_2\text{S}$  could be smelt strongly. This was the case also with South Comstock (Tasmania) ore. Some zinc concentrates from Central Mine (Broken Hill) tailings obtained from an acid float, and agitated in a similar solution to the other tests, showed that the gas above the solution contained  $\text{CO}_2$  2.2 p.c., and oxygen 19.8 p.c.

### Collection and Analysis of Gases evolved from Wetted Minerals.

As it seemed apparent that some gas or gases were still retained on minerals after being wetted with water, it was decided to collect and analyse the gas. This was carried out in the following manner:—The mineral being examined was wetted with distilled water that had been freed from dissolved gases by continued boiling, and was placed in a flask provided with gas-tight connections and connected to a vacuum pump. The flask was immersed in water, which could be heated when required. The gas collected was trapped in portion of the apparatus, and transferred to a gas burette designed to hold and measure small quantities of gas accurately. In the case of minerals, the samples selected were all clean, unaltered and in most cases crystalline fragments, ground to pass an 80 mesh sieve. This material was immersed in air-free water, freed from bubbles of air held mechanically, and then put in the flask as stated above. As some of the minerals floated on reducing the pressure, a small cloth filter was stretched across the end of the joint connecting the capillary tap to the flask. This helped very much in drawing off the gas, as when the sulphides expanded on heating they completely filled the flask, and with the aid of the vacuum pump the cloth acted as a filter, only allowing the gases to pass.

In the case of the products from actual flotation processes the samples of the concentrates were taken from the scum overflowing from the spitz boxes, and put into air-tight bottles in the form of a froth. The bottles were filled as nearly as possible with this scum, together with some of the solution, and kept air-tight till ready for analysis. All the bottles were found to be under reduced pressure. In some cases a sample of the gas above the solution covering the concentrates in the bottles was taken. The results are given in the table B, page (576), and showed that the oxygen content was very low in almost every case. When the zinc concentrates were being tested, the flask was filled as full as possible with the sulphide wetted by the original solution in which they had been floated. After drawing off as much as possible of this solution with a pipette, the flask was filled to the top with air-free distilled water, very little of the original solution remaining in the flask. When the concentrates are taken out of the flask, after being subjected to the reduced pressure, and boiled in water or acid solution, they will as a rule float, this treatment apparently not affecting the flotation properties of the mineral. All the samples in table B would float when heated in their own solution, or if this solution was poured off and water added.

If the sp. gr. of zinc blende is taken as 4, then 4 gms. would displace 1 c.c. of water. Therefore to float 4 gms. of zinc blende 3 c.c. of gas (neglecting its weight) would be required, and 1 gm. would require 0.75 c.c. of gas. On heating this to 100 deg. the volume would be greater, so that if less gas than 0.75 was attached in the cold solution, the mineral would still float on heating.

By collecting the gas evolved in stages it was noticed that the first portion of the gas collected was usually air, and the last to come off was  $\text{CO}_2$ , which apparently showed that the  $\text{CO}_2$  clung more tenaciously to the minerals than did air.

In some cases if the exhausting and heating was continued till the mineral in the flask was perfectly dry, it was found that, even if several c.c. of gas had been trapped in the collecting burette, the gas went back into the flask and could not be again drawn off into the burette unless the mineral was

rewetted. This seemed to show that the dry minerals adsorb  $\text{CO}_2$  and air. A sample of crushed dry crystalline galena was, however, not affected by passing dry  $\text{CO}_2$  over it.

### Description of Apparatus.

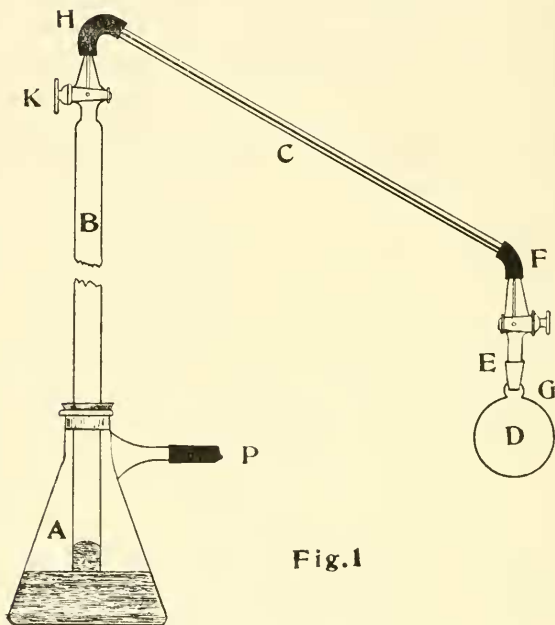


Fig.1

- A—vacuum flask with mercury.
- B—100 c.c. burette 30 inches in height.
- C—capillary tube.
- D—glass gas tight flask 30 c.c. capacity.
- E—tap and tube with ground glass joint.
- F—capillary rubber joints wired on.
- H—do. do. do.
- G—cloth filter on end of joint E.
- K—burette tap.
- P—connection to water pump.

The wetted mineral is placed in flask D, and the tap and tube E is inserted after filling with air-free water. The capillary tube C is filled with air-free water, and wired on to the tap E at F. The burette B is filled with mercury by connecting to the water pump, and then the capillary tube C is wired on at H. A screw clip is also used at H for preventing air getting into tube C when the burette B is disconnected. The flask A is then connected to the water pump at P. On lowering the pressure in A, the gas in the mineral in flask D is drawn over into the burette B, where it can be caught by closing the tap K. The flask D can be immersed in a water bath and heated. The excess of water drawn over into the burette B from the flask D can be drawn off by lowering the pressure in A without allowing any of the gas to escape. Very little gas is lost in solution in the water in this manner, as the water collecting in A is under reduced pressure.

TABLE B.—Gases collected from concentrates from various flotation processes.

No.	Sample	Weight gm.	Gas collected c.c.	p.c. CO <sub>2</sub>	p.c. N	p.c. O	Remarks
1—Zinc concentrates from process using hot acid solution	- - -	60	5.5	81.8			Put into flask without washing. Solution acid to litmus and contained ferrous sulphate
2—Zinc concentrates from process using hot acid solution	- - -	79	0.2	nil			Washed in vacuum filter till free from acid
3—Zinc concentrates from process using hot acid solution	- - -	50	2.4	83.3			Washed free from acid, washed with strong NaOH., then washed free from alkali and added 4 c.c. of a 7.5% H <sub>2</sub> SO <sub>4</sub> solution
4—Zinc concentrates from a film flotation process, collected cold	- - -	49	0.7	28	58	14	This sample taken on different date from Nos. 6, 7, 8. A sample of the gas above the solution in the bottle containing this sample contained: N, 78.5%; O, 19.5%; CO <sub>2</sub> , 2.0%
5—No. 4 sample heated to boiling and collected	- - -	-	2.1	95.2			
6—Zinc concentrates from a film flotation process, collected cold	- - -	46	0.9	nil	74	26	Solution neutral to litmus
7—Zinc concentrates from a film flotation process, collected cold	- - -	49	1.0	25			
8—No. 7 sample, heated to boiling	- - -	49	2.1	95			
9—Zinc concentrates from a vacuum process using oil and acid	- - -	56	1.1	81			Solution acid to litmus



TABLE B.—(Continued).

No.	Sample.	Weight gm.	Gas collected c.c.	p.c. CO <sub>2</sub>	p.c. N	p.c. O	Remarks.
10—	Zinc concentrates from a hot solution oil and acid process	44	8	96.2			25 c.c. of the solution on these concentrates contained 3 c.c. of gas 90% CO <sub>2</sub> . The gas above the solution had the following com- position:—N, 75%; O, 4%; CO <sub>2</sub> , 21%
11—	Another sample from same process as No. 10 test	43	6.4	96			} Gas above the solution: N, 72.5%; O, 2.5%; CO <sub>2</sub> , 25%
12—	Another sample from same process as No. 10 test	46	8	96			
13—	Sample from different vat from same pro- cess as No. 10 test	49	7	94			Gas above the solution: N, 78%; O, 9%; CO <sub>2</sub> , 13%
14—	Zinc concentrates from same process as No. 10 test taken on different date	83	4.3	92			Solution neutral to litmus. Gas above solu- tion: N, 82%; O, 12%; CO <sub>2</sub> , 6%. 75 c.c. of this solution required 1 drop NNa <sub>2</sub> CO <sub>3</sub> to make alkaline with methyl orange
15—	Zinc concentrates from same process as No. 10 test taken on different date	79	3.8	95			
16—	Zinc concentrates from same process as No. 10 test, collected in cold	75	0.6	41.6			
17—	No. 16 sample, heated to boiling	75	4.5	81			

TABLE C.—*Gases collected from different minerals under water.*

No.	Sample	Weight gm.	Gas collected c.c.	p. c. CO <sub>2</sub>	p. c. N	p. c. O	Remarks
1—	Stibnite (Kingwood) - - - - -	60gm	1	23.5	57	19	Sample difficult to wet
	Stibnite (Ringwood) on continuing heating and exhausting - - - - -	-	0.2	100	-	-	The first portion collected was transferred to gas burette and the collection continued
2—	Galena (Mt. Farrel, Tasmania) - - - - -	124	1.1	4.9	45.4	13.6	
	Galena (Mt. Farrel, Tasmania), collection continued - - - - -	-	0.3	100	-	-	
3—	Galena (same sample as No. 2 test, ex- posed to air 14 days) - - - - -	124	3.5	90	-	-	On boiling some of this sample with distilled water a small amount of SO <sub>4</sub> was found in solution
4—	Unoxidised ore from Junction Mine, Broken Hill - - - - -	71	1.3	84.6	-	-	
5—	Pyrrhotite (freshly crushed) - - - - -	63	3.2	73.4	23.2	3.4	
6—	Haematite - - - - -	65	0.6	nil	-	-	
7—	River sand (uncrushed) - - - - -	45	0.5	nil	76	24	
8—	River sand (crushed to pass 120 sieve and boiled with HCl) - - - - -	35	1.2	nil	75	25	
9—	Block 10 Co.'s zinc tailings (coarse) - - - - -	55	0.65	15.3	-	-	After obtaining the float by washing with oleic acid and H <sub>2</sub> SO <sub>4</sub> the concentrates were well washed with H <sub>2</sub> O
10—	Concentrates from a film float from Central Mine tailings - - - - -	30	0.6	83	-	-	
11—	South Mine tailings - - - - -	175	10	85	-	-	
12—	Mount Garnet ore (mixed ZnS and mag- netite) - - - - -	110	5.4	48.1	48.1	3.7	This sample will only give a poor float, but if calneined the float is improved
13—	Mount Garnet ore - - - - -	117	19	90.5	8.2	1.4	Heated to 250° C. for 2 hours
14—	Galena (clean sample of crystalline galena) - - - - -	100	8.5	43	-	-	This sample was difficult to wet and a few bubbles of air were held mechanically

TABLE C.—(Continued).

No.	Sample	Weight gm.	Gas collected c.c.	p. c. CO <sub>2</sub>	p. c. N.	p. c. O.	Remarks.
15—	Galena (clean sample of crystalline galena), exhausted dry	-	0.4	50			This sample was put into the flask dry and heated to 100°C., and the pump connected and exhausted of air. No CO <sub>2</sub> was detected till most of the air had been expelled
	Galena (clean sample of crystalline galena), on further exhausting	-	0.25	90			
16—	Galena (another sample of crystalline galena, Junction Mine, Broken Hill), collected dry	100	8	12			
	Galena (another sample of crystalline galena, Junction Mine, Broken Hill), allowed to wet	-	2.8	100			After exhausting as far as possible the dry mineral, the vacuum was released under air-free distilled water, which was sucked into the flask, and the latter was connected to the pump as before and the gas again collected
17—	Limestone (Marmor, Queensland)	-	0.8	nil	80	20	
18—	Magnetite	-	1.3	nil			
19—	Dry zinc concentrates from an oil and acid float (Broken Hill)	75	2.4	33			
20—	Zinc blende, crystalline specimen exposed to air 24 hours	34	0.7	57			
21—	Granite (orthoclase, quartz and biotite)	25	1.5	52	29	19	
22—	Chalcopyrite	67	1.6	25			
23—	South Mine Lead concentrates, collected dry	117	10	5			
24—	South Mine Lead concentrates, wetted	-	10	100			
25—	South Mine Lead concentrates, collected dry	116	1.5	13			

TABLE C.—(Continued).

No.	Sample	Weight gm.	Gas collected c.c.	p. c. C <sub>02</sub>	p. c. N.	p. c. O.	Remarks.
26	South Mine Lead concentrates, wetted -	-	13	82			This sample of galena was a very pure sample of the mineral. It was crushed through a 60 mesh sieve and boiled with H <sub>2</sub> S for 30 minutes with dilute HCl, much H <sub>2</sub> S being evolved. All soluble salts were then washed out and the galena was exposed to the air for 7 days, and after being wetted with air-free distilled water the gas collected as before
27	Cerussite (Broken Hill) - - -	50	2.7	37			
28	Copper foil cleaned with emery paper -	10	0.5	nil	76	24	
29	Galena from Broken Hill cleaned with HCl	100	0.7	14			
30	Same sample as No. 29 - - -	100	3.7	100			After exhausting the sample used in No. 29 test, the vacuum was released under a 1% acetic acid solution, 7 c.c. being drawn into the flask, which was connected to the pump, and the gas collected as before
31	Sample of galena immersed in kerosene instead of water - - -	105	3.8	8			Remainder of gas was air
32	Broken Hill Proprietary Zinc concentrates immersed in kerosene - - -	60	2.3	7			Remainder of gas was air
33	Cerussite (same sample as No. 27), after standing dry for 4 days - - -	50	5	31			Remainder of gas was air. Sample very difficult to wet and held attached air bubbles
34	Galena (same sample as No. 29), after standing dry for 4 days - - -	87	1.2	41			Remainder of gas was air
35	Coarsely crushed stick sulphur - - -	23	2.1		78	22	

### Summary.

#### *Adsorption of Gases.*

All minerals adsorb gases.

Sulphides appear to naturally absorb  $\text{CO}_2$ .

The consequence of this is—

- (1) Particles are not wholly wetted when immersed in water.
- (2) Particles also tend to float when sprinkled on the surface.
- (3) When immersed in water and heated, the air or gases dissolved in water will collect on the particles, and float or tend to float them to the surface.
- (4) Saturated solutions of gases in water evolve gas which collects on all particles.

*The addition of acids.*—Metallic particles, such as sulphides and metals, when immersed in dilute acid solution, are not wholly wetted, but particles of rock materials become more readily wetted, and give off their adsorbed gas. When different dry minerals are sprinkled on the surface of dilute cold acid, the tendency for them all is to float as in water. The minerals are more readily wetted by hot acid solutions than cold.

*The addition of alkalis.*—When different mineral particles are immersed in an alkaline solution, they tend to part with their adsorbed gases, and will not float even when heated. When sprinkled dry on to the surface of the solution, they remain for a few seconds only, and then break through.

#### *Flotation of Sulphides.*

This is due to the property possessed by minerals of collecting gases on their surface, in such quantity as to diminish the density of the product, to less than that of the liquid. By calculation from the decrease in density it is found that the amount collected on dry sulphides is sometimes half the volume necessary to float them at ordinary temperature. Given a starting point, any gas generated in or introduced into the solution will tend to collect on the surface of the particles. If acid is added, the gases will leave the gangue particles and collect on the sulphides; the latter, therefore, tend to float and the former to remain under the surface of

the liquid. When substances in the solution, such as carbonates, are attacked by acids, the gases evolved saturate the liquid, and commence to grow on the sulphides. Sulphuretted hydrogen, steam, air, and any other gases present either in the solution, introduced into it, or evolved from the minerals present, assist flotation, provided the sulphide particles have dry points on them for the gases to become attached to. Some sulphides naturally adsorb gases, and such sulphides readily float. The bulk of Broken Hill ores may be taken as examples. Other ores require preliminary treatment, so as to alter their surface to some extent. This may be done in certain cases by merely heating to 250 deg. C., or immersing in some solution which will attack the sulphides, such as nitric acid or chlorine solution. Sulphides which condense or adsorb gases well will gather together in a solution as flocculent masses, while those that do not, remain individually distinct, like grains of sand.

In certain cases sulphides will float in acid solution at ordinary temperature, but for the most part with dilute acid solution, flotation starts at about 80 deg. C. The explanation put forward that flotation at that temperature is due to certain carbonates, such as rhodochrosite, siderite, smithsonite being attacked and giving off  $\text{CO}_2$ , does not appear to be correct, for under reduced pressure these minerals are attacked but flotation does not take place. The presence of silicic acid and sulphur, as stated by De Bavay as necessary for a coagulum, is not necessary in many cases of successful flotation. When minerals are treated with dilute acids, and then exposed to the air, certain sulphides adsorb gases on their surface. If they are alternately wetted and exposed to the air, they will become entrapped by the surface film of water which they are brought in contact with. The appearance of the film is the same as that of a bubble of gas under the surface of a solution with sulphides attached. The sulphides in both cases adhere to the surface film of the bubble or free surface of the liquid, but are mainly below the surface itself. The gas obtained in all cases from experimental work and also from samples obtained from various flotation plants was  $\text{CO}_2$ , with varying quantities of nitrogen and oxygen. There is no doubt that

most of this gas is generated from gangue particles in the ore, and being more soluble in the liquid than the other gases, collects readily on the surface of the sulphides.

In all experiments with sulphides carbon dioxide was obtained on exhaustion. This gas must have been present either in the form of a compound easily decomposed by heating or by reducing the pressure, or as a gas condensed on the surfaces of the sulphides, and there retained with greater force than other gases present. It was found that the carbon dioxide was the last gas to come off.

*The effect of oils.*—The effect of oils has long been known in protecting minerals from being wetted by water owing to the oiling of their surfaces, and also the affinity of oils for metals, and many metallic substances. Nearly all the minerals tried could be coated with oil either in the presence or absence of water. This seems to be connected with the adsorption of gases on the particles themselves, for if the particles are deprived of their gas, then as a rule they do not become coated with oil. It would therefore appear that the oiling of particles is dependent on their gaseous attachments. If the particles are oiled to a minute extent, the gases previously adsorbed are retained, and if a solution is heated more gas will become attached to the particles, and flotation will ensue. An oil float may be obtained without acids. Nearly all minerals will float on water after being oiled.

The flotation is apparently due partly to the lesser density of the oil and the presence of gases adsorbed on the minerals or entangled in the oil. If a large quantity of viscous oil is used gases become entangled in it, and the product is oily. In course of time the particles will drop away from it, carrying down attached globules of oil. When a lesser amount of oil is used the product becomes a coherent mass like putty, showing no apparent tendency to float. On further decreasing the quantity of oil, the product becomes firmer, and breaks up into rounded pellets. With a still less quantity of oil, when shaken up with water in presence of air the product becomes flocculent, and increases in volume. This flocculation is due to the adsorption of gases. With very minute quantities of oil the flocculation becomes more pronounced, and the tendency to



float becomes greater. These results may be obtained with gangue and metallic substances. Gases collect more readily on oiled than on unoiled particles.

*The effects of acids on oiled particles.*—The addition of acid causes a more selective action. If oiled gangue and metallic particles are immersed in water, and acid is added, then the oil will tend to leave the gangue particles and become attached to the sulphides. The action of the oil in this respect is similar to that of the gases attached to different minerals, and probably is due to the same cause.

If an ore is treated with an acid and the acid is removed, then if the particles are oiled, the oil tends to become attached to the sulphides only. This is probably due to the removal of the gas from the gangue, which prevents oiling of the same. If acid and oil are added together, the action of the oil is selective, as in the previous case. The presence of oil on their surface does not prevent the minerals being attacked by acids.

The amount of acid required for selective flotation depends on the composition of the ore, the temperature and the pressure existing at the time. Generally it may be stated that an increase of acid will give a purer sulphide product, but flotation will take place at a lower temperature, and with a lesser amount of acid, with oil and acid than with acid alone. A selective flotation can be obtained in some cases by treatment with a chlorine solution previous to the addition of oil, and without the addition of acid.

Some of the light volatile oils will cause flotation in an acid solution at a lower temperature than the heavier and thicker oils. With some Broken Hill tailings a flotation takes place at ordinary temperature with sulphuric acid and a volatile oil.

*Effects of oils and alkalis.*—Flotation can be obtained in alkaline solutions when oil is used, but in every case tried the action was non-selective. On making the solution acid the action becomes selective, only the sulphides floating.

The flotation of mineral particles appears then to depend on the particles having gas attached to them becoming entangled, or caught, by the surface film of a liquid. This film may be (1) the free surface of a liquid, (2) the surface of a

bubble of gas under the surface of a liquid, or (3) the surface of a bubble of gas above the surface of a liquid. Once the particles are entangled in this film they are sustained there. That a considerable weight may be supported by this surface film is shown by the experiment of floating fine-grained mineral on the surface film of water.

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